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EXPERIMENTAL DETERMINATION OF THE MAGNITUDES OF THE ELEMENTAL M--ETC(U)
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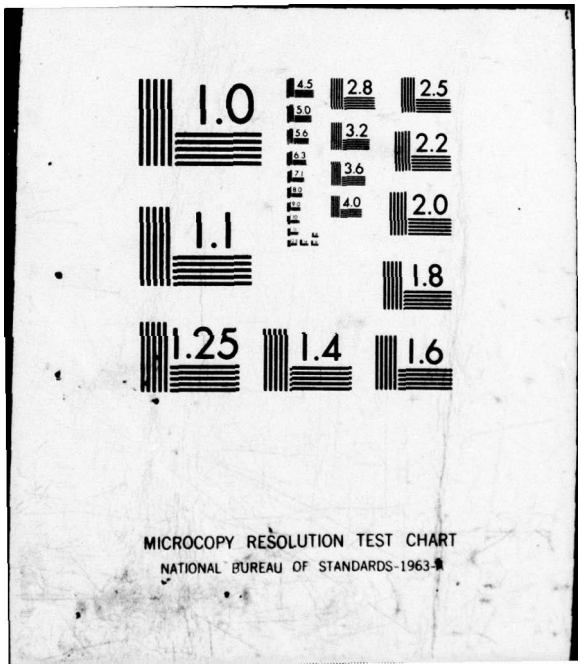
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(Experimentelle Bestimmung der Größe der magnetischen Elementargebiete von Nickel und Eisen. Gerichtete Koagulation von Aerosolen II (1)),

AUTHOR(S): (10) D. Beischer [redacted] August Winkel

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The hypothesis of the elemental magnetic domain

While the characteristics of dia- and paramagnetism can be explained almost completely from the magnetic properties of the single atoms, the characteristics evident with ferromagnetic materials (size, field dependent value of the permeability, appearance of hysteresis phenomena, and a Curie temperature) can be understood only if the mutually alternating effects between adjacent atoms are considered. There are no "ferromagnetic" atoms, but ferromagnetic phenomena appear only when there is a multitude of atoms in a particular geometric arrangement. In the classical theory of ferromagnetism, which was developed essentially by P. WEISS, it is assumed that the moments of the individual atoms of small regions (elemental domains) will act together in the same direction because of the mutual relations between the atoms. The moments of the elemental domain that arise in this way though, cancel themselves out by their mutually random directions in a crystal, so that in the absence of an external field it does not show any magnetic moment. The type of the interaction between the atoms of a ferromagnetic substance was made understandable by W. HEISENBERG (2) as a firm coupling (resonant coupling) between the spins. By assuming an interchangeable magnetic coupling between the spin moments and the spin and orbital moments, F. BLOCH was able to explain homogenous magnetization, hypothetically supposing formation of elemental domains.

However, so far in accordance with this theory only a little can be said about the size and form of these domains. For energetic reasons, the orientation of the unit domains to one another has an oblong form. AKULOV (4) speaks of "filamentous" domains. R. BECKER (5) concludes from the relationship between coercive force and internal tension a size of 100 to 1,000 interatomic distances.

Aggregation of nickel and iron aerosols. Studies below the Curie point.

Because the particular magnitudes fall into the ultramicroscopic area, we have tried to draw upon the working methods of aerosol investigation (6) for a clarification of the problem of the sizes of the elemental magnets. For this purpose we studied the nickel and iron fogs which were produced by thermal decomposition of nickel tetracarbonyl or iron pentacarbonyl in nitrogen. The choice of the carbonyls as starting material has the advantage that at a relatively low temperature the metal aerosols can be produced by condensation from the gaseous phase.

A well purified stream of nitrogen (2 l/hour) was saturated with carbonyl at a certain temperature, and then mixed with a stream of nitrogen (40 l/hour) preheated to 150°. Immediately upon mixing the aerosol which is produced is blown into a sedimentation vessel of 1 l volume. Thus, the flow method has the advantage that the aerosol which arises is exposed to the higher temperature for only a brief time (about 1/4 second). The fogs studied mostly have a concentration of 0.1 to 1 mg/l.

Fig. 1 shows a sediment from a nickel aerosol. The particles consist of long chains that are composed of numerous single filaments. The filaments are about 0.1 mm long and 0.5 μ thick. After some time they become intertwined into small flakes and bundles. In Figs. 2 and 3 sediments

from an older iron fog have formed. The filaments of iron are longer and thinner than those of nickel.

An aggregation that leads to chain-like forms like this is unusual. As a rule, more or less spherical forms arise from aggregation, because each possibility for the orientation of the deposition is equally probable. Previously, we have observed a chain formation only if directional forces acted during the aggregation. Thus we were able to show (1) that small electrical dipoles arose in a pyroelectric substance during cooling from the condensation temperature to room temperature, which form similar chains during the aggregation through mutual electrostatic attraction, as Fig. 1 shows. In nickel and iron aerosols only a magnetic moment comes into the picture, in which case it is of no consequence to the mechanism of the aggregation how this first arises. The following studies will show that it is very probable that during the condensation of nickel or iron fumes that originally arise during the decomposition of the carbonyls small magnets of the order of magnitude of the spontaneously saturated elemental domains are formed and that these particles arise from the mutual attraction of the chains observed. In addition to the directional arrangement of the particles, one support for this point of view is the very great velocity of aggregation. The chains form in fractions of a second, while with clouds consisting of other substances an aggregation period of many seconds is necessary to reach a comparable weight of particles.

Studies above the Curie point

If the explanation given for the aggregation mechanism is correct, chain formation should not take place above the Curie point, because this

is where the large ferromagnetic moments of iron and nickel disappear. Carrying out the studies results in experimental difficulties, because the Curie temperature of nickel (360°) and of iron (770°) are far above the decomposition temperature of the carbonyls (100-150°). Hence, some aggregation with chain formation starts before the metal fog has reached the Curie temperature. A successful setup for this study was one in which we introduced a small amount of liquid carbonyl (0.001 ml) through a gas lock by way of a quartz capillary into a quartz vessel of 35 ml volume open at one side, filled with nitrogen which was preheated to the experimental temperature. Sedimentation of the vapor took place at the temperature of preparation. Fig. 4 shows a sedimentation of a nickel aerosol that was produced above the Curie point at 400°. Only a very few, extremely short chains are visible, along with numerous random, almost modular clumps. At a still higher temperature the chain formation disappears completely. We obtained the same result with iron fogs that were prepared above the Curie point. At temperatures between 800 and 900° only modular shaped aggregates appeared, while below the Curie point the aggregates arranged in the way shown before were obtained immediately. These studies are a direct indication for our assumption of an aggregation shaped by magnetic forces.

Estimation of size of the elemental domains

The size of the smallest particles of which the metal filaments are composed cannot be measured under the microscope because the branched chains observed could be a clumping together of even smaller crystals. Therefore, we have used the method for estimating the size of ultramicroscopic crystallites first proposed by P. SCHERRER, and expanded by M.v. LAUE (7) and

R. BRILL (8). From the broadening of the interferences in the Debye-Scherrer diagram conclusions could be drawn as to the sizes and shapes of crystallites of less than 10^{-5} cm diameter. Fig. 5 shows by diagram and then by the corresponding photometer curves the broad lines for crystallites of nickel aerosol as compared to a similar photograph of a granular crystalline nickel powder. We have found from a series of photographs that the smallest particles of which the nickel filaments consist have a prismatic form. The basal surface of the prism, indicated cubically, is the III-surface. It has a dimension of about 35 x 60 Å (11 x 7 interatomic distances). The length of the prisms is 210 Å (60 interatomic distances)*. The long axis of the

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* The shape of the iron crystallite is given in relation to studies of oxidation processes in the crystallitic surface. Without considering the shape, the iron crystallites have a mean size of 70 Å (25 interatomic distances) (8)

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crystallites has the same crystallographic direction as the direction of the most nominal magnetizability measured by HONDA and KAYA (9). The crystallites primarily formed from the vapor phase have the order of magnitude and shape of the elemental domains given in the Weiss-Heisenberg theory of ferromagnetism, and in the case at hand we can readily identify these directly with them.**

** The sizes for the primary particles that are stated above should not be taken as inflexible quantities for the elemental magnetic domains of nickel and iron. It must be assumed that the elemental magnets become

smaller as the temperature increases, and that also they can increase during homogenous magnetization of a large crystallite.

These then collect free moments and accumulate into the chains observed in the sediments.

Spontaneous magnetization of the elemental domains

When aggregation takes place under the influence of the magnetic earth field a magnet moment can be induced in the primary particles by this field, which would lead to the same ordered aggregates as those we have observed. In order to indicate that the moment of the elemental domains appears spontaneously, or in other words, without any external field effect, we have tried to exclude the earth field during the formation and aggregation of the aerosol as much as possible. However, so far we have been able to reduce its strength in the experimental container only to 0.004 Oersted. A field like this would probably be sufficient for a constrained magnetization of the particles. The fact that under these conditions an ordered aggregation occurs can still not be regarded as conclusive evidence for the spontaneous appearance of moments in the elemental domains, as it is required by the theory of ferromagnetism.

Properties of the aerosol particles in the magnetic field

Under the ultramicroscope the particles of a nickel or iron fog appear to be a series of luminous points like a string of pearls, which bend and curve in a varied way because of the Brownian movement. If a weak magnetic field of about 3 Oersted is imposed on the test cell, then the filaments immediately arrange themselves with their long axis in the direction of the

field. When the poles are reversed, they rotate about by 180°. The resistance to a magnetic reversal is also larger than the force required for a mechanical rotation of the particles in the gas. Unfortunately, quantitative measurements of the rotatory velocity of the particles by the Brownian movement are very difficult to make.

Legend for Figures

Fig. 1 Sediment from a nickel aerosol prepared below the Curie temperature. (Magnification x 300)

Fig. 2 Sediment from an aged iron aerosol with floc formation, prepared below the Curie temperature. (Magnification x 180)

Fig. 3 Sediment from an aged iron aerosol with strand formation, prepared below the Curie temperature. (Magnification x 300)

Fig. 4 A sediment from a nickel aerosol prepared above the Curie point, showing no chain formation. (Magnification x 300)

Fig. 5 Debye-Sherrer diagram with photometer curve for a nickel sediment with the crystallite size 200 x 50 x 35 Å (a) compared with the corresponding diagrams for granular crystalline nickel powder (b).